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### FIELD-EMITTER ARRAYS FOR **RFVACUUM MICROELECTRONICS**

C.A. Spindt, Program Director C.E. Holland, Sr. Research Physicist P.R. Schwoebel, Research Engineer Physical Electronics Laboratory

SRI Project 2743

Prepared for:

Advanced Research Projects Agency Defense Sciences Office Virginia Square Plaza 3701 North Fairfax Drive Arlington, VA 22203-1714

Attn: Dr. Bertram H. Hui

ARPA Order No. 8162

Covering the Period 1 July through 30 September 1993

Contract MDA972-91-C-0029

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#### 13. ABSTRACT (Maximum 200 words)

SRI International has completed the eighth quarter of a program to develop field-emitter arrays for vacuum microelectronics. We have met the first-phase program goals of 5 mA total emission, with a current density of 5 A/cm<sup>2</sup> for at least 2 hours and demonstrated modulation of the emission current at a frequency of 1 GHz.

A two-step reactive-ion-etch process was established to eliminate an earlier problem with sulfur contamination during low-capacitance cathode fabrication. An electrode designed to shield the emission area from the fringing electrostatic field caused by the proximity of the base electrode in the low-capacitance configuration was fabricated and tested. Measurements continued on one of four cathodes set up for test in a high-frequency apparatus.

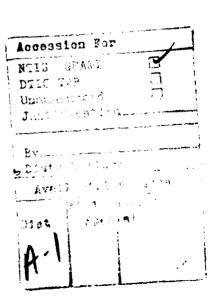
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#### **EXECUTIVE SUMMARY**

SRI International has completed the eighth quarter of Phase I of a research and development program on the SRI Spindt-type field-emitter-array cathode with a view toward eventual applications in microwave amplifiers. We have met the first-phase goals of 5 mA total emission, with a current density of 5 A/cm<sup>2</sup> for at least 2 hours and demonstrated modulation of the emission current at a frequency of 1 GHz. Our approach has been to identify methods of adapting and modifying the basic cathode structure of microwave operation and to experimentally investigate means of implementing those methods.

During the quarter we have accomplished the following, as documented in detail in this technical report:

- Continued research on basic cathode technology as defined by the goals of the ARPA program and related NRL project (Section 1)
- Determined the probable cause of sulfur contamination during low-capacitance cathode fabrication and established a two-step reactive-ion-etch process with SF<sub>6</sub> and CF<sub>4</sub> to eliminate the problem (Section 2)
- Fabricated and tested the electrode designed to shield the emission area from the fringing electrostatic field caused by the proximity of the base electrode in the low-capacitance configuration (Section 3)
- Continued measurements on one of the four cathodes set up for test in a high-frequency apparatus (Section 4)
- Planned activities for the period of 1 October through 31 December 1993 (Section 5)



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#### 1. INTRODUCTION

SRI International is participating in an effort of the Advanced Research Projects Agency (ARPA) and the Naval Research Laboratory (NRL) to perform research and development on the SRI Spindt-type field-emitter-array cathode with a view toward eventual applications in microwave amplifiers. The current ARPA program is the vehicle for advancing the basic cathode technology for microwave applications (e.g., reducing intrinsic capacitance and driving voltage requirements), and continues the original program plan to establish the characteristics of the cathode in its preprogram state of development, identify methods of adapting and modifying the structure for microwave operation, and experimentally investigate means of implementing those methods. For the NRL program, which began earlier than the ARPA project, SRI has shifted emphasis to the support of NRL's in-house vacuum microelectronics program by providing NRL with state-of-the-art Spindt-type cathodes and consultation on setting up and using cathodes. We have met the first-phase program goals of 5 mA total emission, with a current density of 5 A/cm<sup>2</sup> for at least 2 hours and demonstrated modulation of the emission current at a frequency of 1 GHz.

At the beginning of the program, two areas of development required immediate attention. The first was a materials and processing issue related to providing and maintaining a suitable vacuum environment for the cathodes. The second related to the cathode's inherent high capacitance and means for reducing that capacitance to a level that is consistent with the microwave applications envisioned for the cathode.

Our approach has been to research these two issues in parallel, using an easy-to-build, low-frequency-triode configuration fabricated on a TO-5 header as a test vehicle for materials and processing studies, and at the same time designing and researching fabrication techniques for building high-frequency-cathode structures on dielectric substrates (e.g., quartz or glass). Specific tasks that are being addressed on these related programs are

- 1. Fabrication of a supply of state-of-the-art cathodes for use in establishing cathode characteristics, and for developing structures, circuits, and procedures for testing the cathodes as triodes
- 2. Development of a close-spaced anode test configuration that can be used to investigate triode characteristics at low frequency (kHz to MHz) in order to study the known problems with cathode survival under close-spaced anode conditions
- 3. Development of a circuit for driving the cathodes and demonstrating gain, frequency response, and peak emission levels
- 4. Studies of advanced cathode structures (geometry, fabrication technology, and processing) for high-frequency operation

- 5. Investigations (with NRL) of cathode mounting and connecting procedures using practices that are consistent with the microwave goals of the effort
- 6. Consultations with the NRL staff on the experimental results and applications of the cathode technology

#### 2. LOW-CAPACITANCE CATHODE FABRICATION

A series of carefully controlled tests combined with Auger spectroscopy positively identified a persistent and ongoing contamination problem as being due to sulfur contamination from our reactive ion etcher. We had changed our reactive-ion-etch (RIE) gas from CF<sub>4</sub> to SF<sub>6</sub> in order to pattern the gate electrode. This change was necessary because the CF<sub>4</sub> etched away the photoresist before the patterning of the chromium was completed. We determined, however, that even though the SF<sub>6</sub> completely etched the chromium well before the resist was significantly damaged, the SF<sub>6</sub> left a residue of sulfur on the edge of the etched molybdenum gate film under the resist where it could not be detected by the Auger spectrometer. When the substrate was heated during subsequent processing, the sulfur migrated over the entire surface of the cathode and caused erratic emission behavior and cathode failures. We are avoiding this problem by etching most of the gate pattern with the relatively fast SF<sub>6</sub> and then finishing the process with the CF<sub>4</sub>, which removes the sulfur residue while completing the gate pattern etch. This two-step RIE process has now been adopted as our standard gate-film-patterning procedure.

Our resolution of the sulfur contamination problem enabled us to resume fabrication and testing of the low-capacitance cathodes late in the quarter Earlier in our research, we had determined that the emitter-tip/gate geometry is a very important parameter in determining the current/voltage characteristics of the emitter array. Generally, smaller aperture diameters and taller, sharper emitter tips are preferred for low-voltage, high-transconductance performance. Figure 1 is a scanning electron micrograph of an early (precontamination era) emitter array that performed very well. However, recent cone formation runs using the same apparatus and procedures have produced inconsistent results and less desirable cone shapes, as illustrated in Figure 2. These variations in results were first noticed while the more important task of solving the contamination problem dominated our efforts. Now that the contamination seems to be under control, we will concentrate on the issue of inconsistent hole/cone geometry.

#### 2.1 AUGER SPECTROSCOPY RESULTS

The Auger studies made during mid quarter examined cathodes 103-E4-4S, 54E-316-1U (both of which had been tested), and 101L-A5-1B (which had not been tested). All the cathodes were stored in pinch-off tubes, and had therefore been vacuum baked.

Cathode 103-E4-4S showed a monolayer to submonolayer coverage of sulfur present on the surface of the gate film and gold pads. Carbon, nitrogen, and oxygen were also observed. These contaminants are to be expected following atmospheric exposure. Brief argon sputtering removed the sulfur contamination, indicating that it was present only on the surface. Subsequent depth profiling indicated that the gate film was roughly stoichiometric molybdenum carbide in the form of Mo<sub>2</sub>C at least to a depth on the order of 100 monolayers. Profiling was not continued beyond this depth.

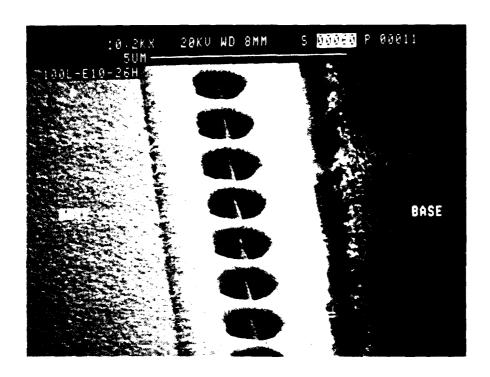
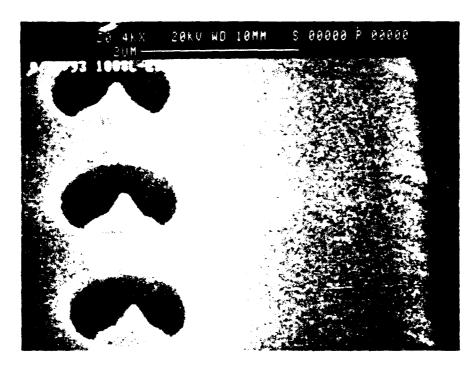


Figure 1. Scanning electron micrograph of an early low-capacitance cathode with excellent cone height and shape



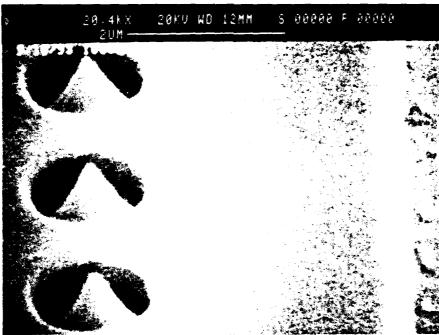


Figure 2. Scanning electron micrographs of two low-capacitance cathodes showing varying, and less than optimum, cone shapes

Cathode 54E-316-1U showed no sulfur contamination on the surface; depth profiling showed a gate film composed of Mo<sub>2</sub>C. Cathode 101L-A5-1B had no surface sulfur contamination, but depth profiling revealed the presence of a Mo<sub>2</sub>C gate film.

Because the primary difference in the three cathodes was the use of SF<sub>6</sub> in the preparation of the gate and base films for cathode 103-E4-4S, investigations were conducted to determine whether the SF<sub>6</sub> led to the sulfur contamination. We especially noted that molybdenum forms a very stable sulfide.

We conducted tests in which molybdenum films were patterned, then etched completely through with SF<sub>6</sub>, and then with SF<sub>6</sub> followed by CF<sub>4</sub>. In both cases, no sulfur was detected on the molybdenum gate or on the substrate. To investigate the possibility that the sulfur was present at the edges of the molybdenum film (it could subsequently migrate over the surface during vacuum baking at ~700 K) molybdenum films were only partially etched through with pure SF<sub>6</sub>, and then with SF<sub>6</sub> followed by CF<sub>4</sub>. With the pure SF<sub>6</sub> etch, sulfur was found on the surface of the film. When the SF<sub>6</sub> was followed by an etch with CF<sub>4</sub>, no sulfur was present; a small amount of fluorine was detected. Since molybdenum does not form a very stable fluoride, this was considered inconsequential.

Depth profiling again revealed both films to be composed of  $Mo_2C$ . The mobility of sulfur on  $Mo_2C$  should be very high compared to its mobility on pure molybdenum, since  $Mo_2C$  is a more stable compound than  $Mo_xS_y$  for any x and y.

#### 2.2 SIGNIFICANCE OF MOLYBDENUM CARBIDE

The presence of Mo<sub>2</sub>C in the gate film is very important, since the procedures and apparatus used to deposit the gate film are the same as those used to deposit the emitter cones. Thus, it may have been that our emission was obtained from Mo<sub>2</sub>C rather than Mo.

The Auger electron spectroscopy observations are consistent with the general chemical behavior of the films. We have noticed that the tips and gate films are not attacked by H<sub>3</sub>PO<sub>4</sub>. This is in agreement with the literature, which indicates that Mo is attacked by H<sub>3</sub>PO<sub>4</sub>, whereas Mo<sub>2</sub>C is quite resistant to attack.

It is clearly quite important that we understand and control the extent of carbide formation, as this is critical to many performance issues, including the *enhancement* of emitter operations and reliable creation thereof. Because these issues are outside the scope of the present NRL and ARPA microwave cathode development programs, we may have to find new funding to pursue the investigation.

#### 3. EMISSION TESTS

Early in the quarter, we fabricated and tested the electrode designed to thield the emission area from the fringing electrostatic field caused by the proximity of the base electrode in the low-capacitance configuration. Figure 3 shows the configuration, and Figure 4 shows the results very clearly. Figure 4a indicates the emission current and gate current with the shield electrode at base potential, and Figure 4b illustrates the same data with the shield at gate potential. The anode was at +400 V in both cases.

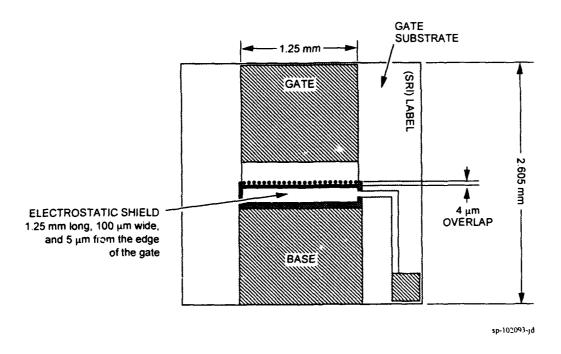
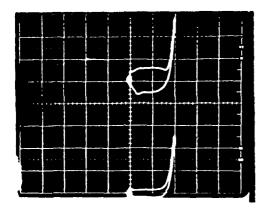
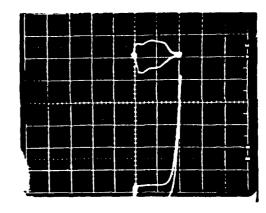


Figure 3. Schematic diagram of the location and relative size of the base shield





(a) With shield at base potential

(b) With shield at gate potential

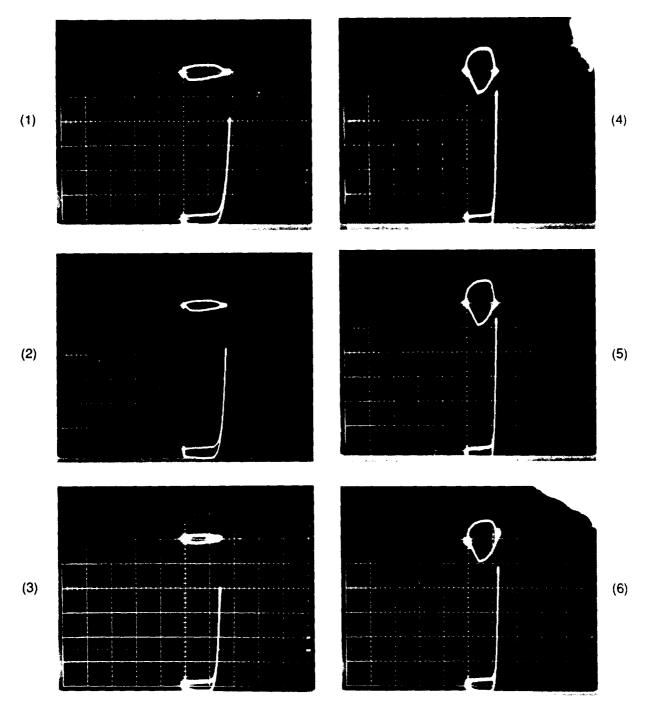
Top trace = gate current = 10  $\mu$ A/division Bottom trace = emission current = 200  $\mu$ A/division Horizontal = 50 V/division

Figure 4. Current/voltage curves for cathode 103L-E10+2A showing the effects of the base shield on gate current

This shield was also shown to function well as an anode, suggesting that a monolithic triode structure of the kind first described by Geppert (U.S. Patent 3,701,919: October 1973) and later reported with field-emission cathodes by Gray, Campisi, and Greene of NRL (*IDEM Digest:* 1986) might well be worth investigating with this geometry. A brief test with the shield at +160 V, anode at -22 V, tips at ground, and gate at 130 V showed that with a total emission of 100  $\mu$ A, 80  $\mu$ A was collected at the shield electrode. A possible triode configuration is described in Section 3.2.

#### 3.1 TEST RESULTS

After CF<sub>4</sub> was determined to be an appropriate RIE for the gate and base-shield electrodes, emissions were reinitiated with a new group of low-capacitance cathodes and a group of standard cathodes (10,000 tips) on silicon as a control. Figure 5 shows the initial current/voltage characteristics at 10 µA peak emission shortly after first turn-on. The cathodes were mounted on TO-5 headers and were all in the same six-position test site, which is a standard test chamber that we have been using for several years. The chamber was baked at 700 K for 72 hours and then cooled to 350 K before the cathodes were turned on. The background pressure was in the mid 10<sup>-9</sup> torr range at turn-on. The purpose was to bring the cathodes up to normal operating levels and compare their behavior. We hoped that the low-capacitance cathodes would behave in the same way as the standard cathodes, thus indicating that the contamination problem had been solved. Low-capacitance cathodes that suffered from the sulfur contamination exhibited erratic emission and, with time, a steady fall in emission level until they were no longer operational.



Horizontal: 50 Volts/Major Division; Vertical: 5 µA/Major Division

- (1) Low-capacitance cathode 103L-E11+5B (250 tips)
- (2) Low-capacitance cathode 102L-E11+5D (625 tips)
- (3) Low-capacitance cathode 102L-E11+5I (625 tips)
- (4) Standard cathode 52i-344-4C (10,000 tips)
- (5) Standard cathode 52i-344-4D (10,000 tips)
- (6) Standard cathode 52i-344-4F (10,000 tips)

Figure 5. Initial current/voltage characteristics for three low-capacitance cathodes and three standard 10,000-tip cathodes on silicon operated at the same time in the same test chamber for comparison

During mid quarter, emission was initiated with the test group, and brought up to  $10 \,\mu\text{A}$  per cathode. Later, the emission was increased to  $100 \,\mu\text{A}$  on all the cathodes. Drive voltage producing that level of emission was held constant for two weeks so that we could determine if emission would remain stable over time. At the end of this period, we observed that emission was essentially unchanged on all six cathodes.

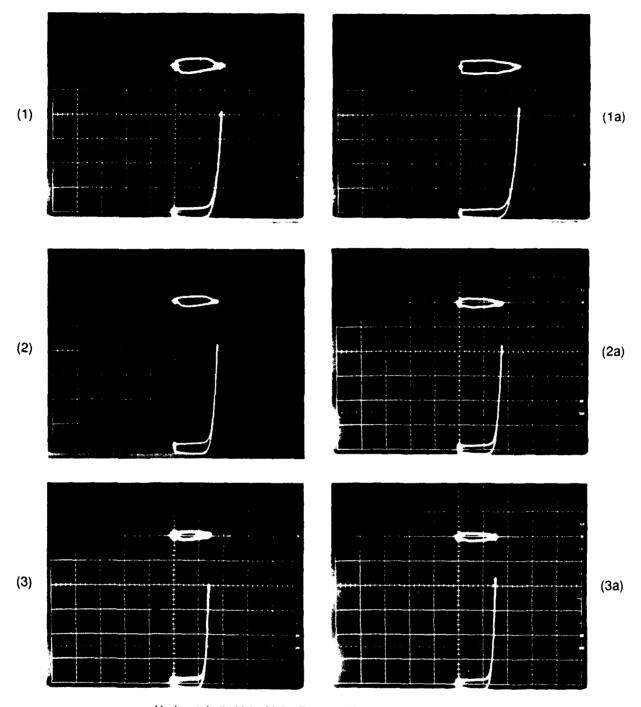
Figure 6 shows the current/voltage characteristics of the three low-capacitance cathodes at  $10 \,\mu\text{A}$  peak emission after two weeks of operation at  $100 \,\mu\text{A}$ , and the current-voltage characteristics taken at the beginning of the test period for comparison. There is very little change in the characteristics. This is very encouraging and suggests that the contamination problem has indeed been solved. During the next period, the emission levels will be increased.

#### 3.2 PROPOSED TRIODE CONFIGURATION

A schematic diagram of a proposed monolithic triode structure fabricated on a diamond substrate is shown in Figure 7. The heat-sinking substrate could lead to a compact, relatively high-power triode (25 mA has been achieved; 500 V on the anode would result in a 12.4-W device). The base electrode is routed out the sides rather than under the shield to reduce capacitance; the present shield is used as an anode.

The thicker anode would improve the electrostatic field distribution with respect to overcoming space charge effects in the immediate vicinity of the emitter tips, and would enhance the collection efficiency of the anode.

Figure 8 is a plan view of the proposed triode, showing the microstrip line configuration of the gate and anode electrodes and the contact pads for grounding the base electrodes.



Horizontal: 50 Volts/Major Division; Vertical:  $5 \mu A/Major$  Division

(1 and 1a) Low-capacitance cathode 103L-E11+5B (250 tips)

(2 and 2a) Low-capacitance cathode 102L-E11+5D (625 tips)

(3 and 3a) Low-capacitance cathode 102L-E11+5I (625 tips)

Figure 6. Current/voltge characteristics for three low-capacitance cathodes at the start of emission (1, 2, and 3), and after two weeks operating at 100  $\mu$ A and then turned back to ~20  $\mu$ A for comparison to the original performance (1a, 2a, and 3a)

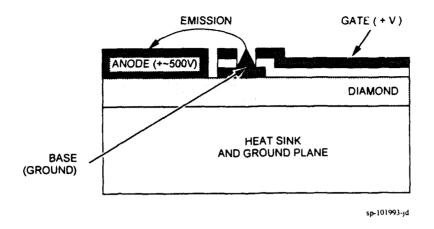


Figure 7. Monolithic triode structure on a diamond substrate with a heat sink

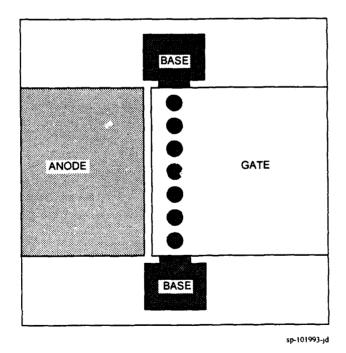


Figure 8. Plan view of a monolithic triode on a diamond neat-sink substrate

#### 4. HIGH-FREQUENCY MEASUREMENTS

In June, four cathodes were placed in our high-frequency test apparatus as we attempted to produce test data for the annual vacuum electronics review. The cathodes were somewhat inferior because we had not yet completely eliminated the sulfur contamination. Nevertheless, one of the cathodes was carefully nursed into decent behavior. (The other three failed early because of erratic operation, probably resulting from the contamination.)

Measurements of S21 were made at 1, 3, and 5 mA with the good cathode. S parameter measurements above 5 mA were not possible because the network analyzer requires that the cathode be ON for at least 1 second for a measurement to be made, and with a 1-second ON time and 20-second OFF time the anode became orange hot at the end of the 1-second ON time. However, enough data were taken to show that the transmission coefficient (S21) is 3.5 dB greater at 5 mA and 1 GHz than it is at <1  $\mu$ A emission. Figure 9 shows the relative magnitudes of S21 for three values of emission.

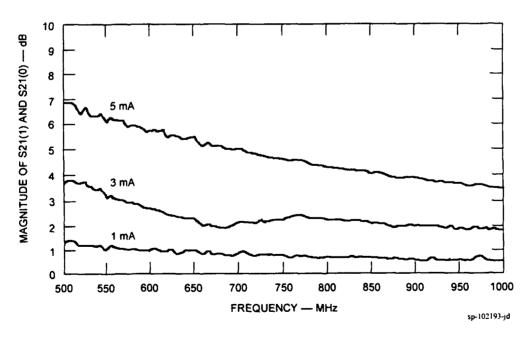


Figure 9. Magnitude of S21 at 1-, 3-, and 5-mA emission levels relative to <1 μA emission for cathode 102L-E10+5C

Figure 10 shows the results of attempts to measure S11 and S22 with the apparatus we have. Clearly something (probably our calibration standard) is not quite right, because the forward reflection coefficient cannot be greater than 1 (0 dB). We worked with network analyzer specialists in an effort to improve these results.

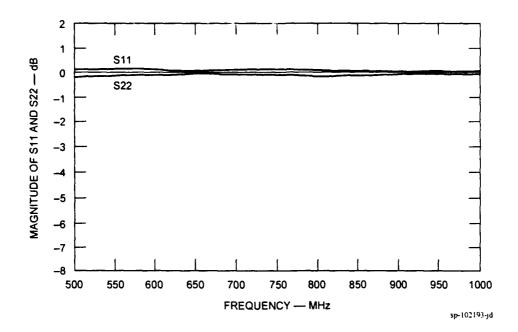


Figure 10. Measured forward and reverse reflection coefficients for cathode 102L-E10+5C showing an obvious error in the S11 parameter, probably due to an imperfect calibration standard

At mid quarter, high-frequency measurements were discontinued pending the results of tests with low-capacitance cathodes fabricated using procedures designed to eliminate the sulfur contamination problem.

#### 5. WORK PLANNED

Attempts to improve performance through processing and fabricating smaller geometries will continue. Additional high-frequency triode measurements will be made as devices become available, and strong efforts will be made to provide an adequate supply of cathodes in support of NRL's work.

Tests with *in-situ* processing will be continued to determine methods of improving emission uniformity and reducing operating voltage requirements. Emission levels will be increased in further tests to verify the elimination of the contamination problem. The issue of inconsistent hole/cone geometry will be addressed.